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JOURNAL OF
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The Official Organ of The American Oil Chemists' Society

Vol III SEPTEMBER, 1926 No. 9

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VOL. III **SEPTEMBER, 1926** **No. 9**

EDITORIAL

THE GREATEST OF COMPETITIONS

On another page in this issue of the JOURNAL, the Chairman of the Ammonia Committee calls attention to the Smalley Foundation Cup. The competition for this prize should be one of the most cherished possessions of the American Oil Chemists' Society. Both for accuracy and in respect to the number of competitors interested, the work probably ranks second to no other similar undertaking on the part of any chemical society or group in this country.

The Smalley Foundation was instituted by the late Frank N. Smalley to determine the best methods for analyzing cottonseed meals for oil and ammonia. Originally it was organized in the Southern Cotton Oil Company in order to have each district laboratory check against the other. Today this has developed into a check contest of about eighty laboratories. Each laboratory analyzes one sample a week for thirty weeks, and every Monday they report the results of the oil, ammonia and moisture tests.

The original Smalley Cup now rests in the permanent possession of the Battle Laboratory; but owing to the generosity of Dr. H. B. Battle a new cup has been provided. On September 20, the 1926-1927 competition begins. This prize is well worth a man's best efforts and it is to be

hoped that the competition this year will bring out even more contestants and result in still higher standards of laboratory excellence.

THE CHEVREUL PRIZES

We apologize to the shades of the great CHEVREUL for having messed up his name in our August issue. A malicious interpretation of what really was a typographical error would be to accuse the Prize Committee of pulling a publicity stunt, and this view would have been substantiated by the correspondence the misprint has since evoked. Many readers of the JOURNAL have "written to the editor"; but we are very happy to announce that not in a single instance was cancellation of a subscription threatened.

The announcement of these prizes is of great importance to one branch of the oil and fat industry just at this particular time. The new season for the cottonseed crushers is upon us. In the many activities that will take place among the cottonseed oil men during the coming months, an opportunity is afforded for observation and experimentation that will be invaluable to them in competing for the prizes.

Remember! Ginners were made subject to the so-called Pure Food Law by Regulatory Announcement 261 of the Bureau of Chemistry, issued in 1918. Returning to cottonseed the foreign matter that has been removed in the ginning process is a violation of the Federal Food and Drug Act.

CRUDE OIL ANALYSIS

By DAVID WESSON

The discussions at the 1926 meeting of the American Oil Chemists Society have shown that the time is now ripe to discard the refining test, so long used in determining the value of crude vegetable oils, and resort to scientific methods of chemical analysis.

There was a time when iron masters bought iron ore for their furnaces on what was known as the crucible assay. A known weight of ore, limestone and powdered charcoal or coke were heated in a Hessian crucible in a coke fire for several hours, after which the crucible was withdrawn, cooled, and broken, and a button of iron, separated from the slag and weighed. This was taken as the amount of iron present in the ore. Like our refining test it was uncertain in results, depending a good deal on the skill of the operator and the character of the ore. At the best, it was a rough test, and perhaps good enough for the primitive plants of its day.

With the advent of the chemist in the iron industry, it was found possible to determine exactly how much iron was contained in an ore, and the character of impurities which would influence the yield in the furnace, and the quality of the product. This information put a correct value on the ore. There is no logical reason why such methods should not apply to the valuation of crude cottonseed oil.

In 1916 and 1917, the writer, working on the problem of improving refining methods, concluded that the first step was to find out how to analyze the oil so as to have some basis for getting at the efficiency of refining operations and the causes which influenced the losses.

With the assistance of Harold P. Gaylord, a method was worked out in the writer's laboratory. It was tested in other laboratories, and several years after was carefully checked up by Dr. Geo. P. Jamieson, of the U. S. Bureau of Chemistry. The method shows, absolutely, the amount of refined oil present in a given sample of crude oil, and gives a standard by which a refining operation or method can be judged.

In applying it to commercial practice, allowance would naturally have to be made for the efficiency of refining operations in the plant, in getting at the value of the oil to the purchaser. If the refiner does not work to a high state of efficiency, that is his own misfortune; not the fault of the man who sells the crude oil. If the man who sells the crude oil, pumps water and settlings into it, the result will be apparent in the sample when analyzed.

The following description of the test is given as written in 1917, and the numerous examples of its application bear tribute to its accuracy:

Absolute Method for Oil Analysis

The object of this method is to determine the maximum amount of refined oil which can be produced from a given sample of crude or partly refined oil and at the same time to determine the amount of fat necessary to saponify and the amount of caustic soda necessarily absorbed in the refining of the oil.

It has been found as the result of careful experiment that when an oil is dissolved in gasoline the mixture of oil and gasoline can be shaken up with a large excess of caustic potash, which will combine with the coloring matter, free fatty acids, and break up the lipoid bodies in the cold inside of 3 minutes. By adding a considerable quantity of 50 per cent alcohol and water the excess caustic, together with the soap and products of decomposition of the organic matter, color, etc., settles out, leaving a clean gasoline solution of refined oil which can be readily separated from the solution of soap-stock.

After washing all oil out of the alcohol solution of soap-stock there is no difficulty in running the latter down to dryness and determining the fatty acids therein by following the regular procedure. It is also possible by this method, operating on a separate weight of oil, to determine the alkali absorbed, which is done by titrating a blank representing the caustic potash started with and titrating the excess alkali in the alcohol soap solution, using phenolphthalein as an indicator.

APPARATUS:

A quantity of 4-ounce oil sample bottles.
A wash bottle blow-off arrangement such as is used for soap-stock tests.
Soxhlet flasks.
Erlenmeyer flasks.
A pipette graduated to deliver 10 grams of oil at 20° C.
Acid and alkali burettes.

REAGENTS:

Redistilled gasoline, less than 80° B. P. Cent.
Fifty per cent grain alcohol. (Denatured will not do.)
Caustic potash, 14 per cent solution. (Caustic soda will not do.)
Phenolphthalein.
Quarter or half normal HCl.
Quarter or half normal NaOH.

PROCESS: Pipette 10 grams of a well shaken sample into a small tared beaker and then take the exact weight of the oil. Wash the oil into a 4-ounce sample bottle, using 50 cc. of gasoline for the purpose. Pipette 10

cc. KOH solution into the well mixed oil and gasoline and shake for 3 minutes. Then measure in 25 cc. 1 to 1 alcohol and shake vigorously until the soap separates easily. In some oils the separation is almost instantaneous: in other oils it is very slow. As soon as a good separation is shown, which should be in less than a minute, whirl in centrifuge and let stand a few minutes before blowing off the gasoline solution of the oil into a clean tared Soxhlet flask. Shake up twice, using 20 cc. of gasoline each time. Blow off the same after each washing into Soxhlet flask. Time can be saved by whirling after each addition of the gasoline. Distill off the

Table No. 1.—Showing Possible Accuracy of Absolute Oil Test

Samples	Absolute Oil A	Absolute Oil B		Absolute Oil A	Absolute Oil B
Sav. 39-a	96.79	96.82	N. O. No. 2	96.95	97.15
" 35-b	97.05	97.10	Mem. 54-A	96.57	96.57
" 43	97.	96.80	Check No. 1	96.75	96.65
" 44	96.95	96.97	" " 2	96.90	96.87
" 34	96.03	96.20	" " 2 (Centrifugal)	97.17	97.27
" 40-b	96.73	96.65	B. Comp. No. 5	97.23	97.39
" 37-e	94.05	94.			
" 41-d	96.83	96.95			
" 36-a	96.79	96.85			
" 38-c	97.19	97.32			
B 312	97.05	97.07			
" 310	95.79	95.55			
" 309	97.20	97.32			
" 317	97.73	97.63			
Sav. Comp. No. 1	96.97	96.90			
N. O. " " 1	96.75	96.57			
Sav. " " 2	96.65	96.60			
Memphis No. 2	97.33	97.30			

Table No. 2.—Results Obtained by Applying the Absolute Test to Refined Oils

Oil	Absolute Oil	Absolute Loss	Moisture	F. F. A.	
B. 312	99.80	.20	.16	.06	.22
B. 309	99.78	.22	.14	.05	.19
B. 311	99.83	.17	.18	.07	.25
B. 317	99.79	.21	.16	.04	.20
B. 310	99.75	.25	.18	.03	.21
N. O. 110	99.80	.20	Present but not determined		
Deodorized Oil	99.84	.16	" " " "		

Table No. 3.—Method of Reporting Results

No. Refinings	Memphis No. 8	Bayonne No. 11	N. O. No. 7	Sav. No. 8
Date	3/17/17	3/12/17	3/3-3/9/17	No Date
Absolute Oil	96.85	97.07	96.25	95.49
Absolute Loss	3.15	2.93	3.75	4.51
F. A. as Soap	1.58	1.55	1.95	2.95
Loss Not Fat	1.57	1.38	1.80	1.56
NaOH Absorbed	.365	.365	.415	.555
NaOH for F. F. A.	.160	.172	.235	.365
F. F. A.	1.13	1.21	1.66	2.58
F. A. as Soap—F. F. A.	.44	.34	.29	.37
Color Drops	Slowly	Slowly	Quickly	Quickly

Table No. 4.—Showing Accuracy of Checks When Determining Absolute Oil

12 Bayonne Refinings

Number	1 Absolute Oil	2 Absolute Oil	Difference
159	98.10	97.90	.20
160	97.45	97.50	.05
161	97.67	97.43	.24
162	97.39	97.56	.17
163	97.70	97.66	.04
167	97.83	97.77	.06
177	97.27	97.20	.07
173	97.50	97.60	.10
175	97.27	97.20	.07
178	96.99	96.93	.06
169	97.73	97.65	.08
168	97.90	97.80	.10
12	Average		.103

8 New Orleans Refinings

Number	1 Absolute Oil	2 Absolute Oil	Difference
92	96.83	96.73	.10
97	97.47	97.47	.00
106	96.60	96.55	.05
110	97.63	97.67	.04
111	97.07	97.07	.00
113	98.40	98.29	.11
124	96.47	96.35	.12
91	96.90	96.97	.07
8	Average		.06

8 Memphis Refinings

Number	1 Absolute Oil	2 Absolute Oil	Difference
76	96.99	96.99	.00
2	98.67	98.67	.00
55	97.23	97.30	.07
1	97.89	97.37	.23
3	98.64	98.84	.20
73	97.00	97.30	.30
75	98.65	98.55	.10
77	97.39	97.43	.04
8	Average		.117

Table No. 5 (a)

Analyses of Bayonne Refinings

Number	Absol. Oil	Absol. Loss	Alk. Abs.	F. S. as S.	Loss not Fat	F. F. A.
159	98.00	2.00	.30	1.10	.90	1.00
160	97.47	2.53	.36	1.51	1.02	1.10
161	97.55	2.45	.28	1.40	1.05	.90
162	97.47	2.53	.30	1.28	1.25	1.00
163	97.68	2.32	.25	1.51	.83	1.00
177	97.23	2.77	.51	1.73	1.04	1.30
173	97.55	2.45	.36			.90
175	97.23	2.44	.40	1.45	1.32	.90
178	96.96	3.04	.45	2.25	.79	1.60
169	97.69	2.31	.38	1.68	.63	1.20
168	97.85	2.15	.30	1.40	.75	1.00

Table No. 5—(Continued)

Application of Analyses

Number	F. F. A.	Absol. Oil	Absol. Loss	Kettle Loss	Kettle Yield	Efficiency
159	1.00	98.00	2.00	4.91	95.09	97.
160	1.10	97.47	2.53	6.21	93.79	96.2
161	.90	97.55	2.45	6.66	93.34	96.
162	1.00	97.47	2.53	6.62	93.38	95.8
163	1.00	97.68	2.32	6.20	93.80	95.
177	1.30	97.23	2.77	6.32	93.68	97.
173	.90	97.55	2.45	4.93	95.07	97.4
175	.90	97.23	2.77	6.01	93.99	97.
178	1.60	96.96	3.04	7.09	92.91	95.8
169	1.20	97.69	2.31	6.64	93.36	95.6
168	1.00	97.85	2.15	4.35	95.65	97.8
Average	1.08	97.52	2.48	5.99	94.01	96.51

Table No. 5 (b)

Analyses of Memphis Refinings

Number	Absol. Oil	Absol. Loss	NaOH	F. A. as Soap	Loss not Fat	F. F. A.
76	96.99	3.01	.34	1.35	1.66	.7
2	98.67	1.33	.24	1.11	.22	.9
55	97.26	2.74	.29	1.62	1.12	1.00
1	98.00	2.00	.25	.79	1.21	1.00
3	98.74	1.26	.20	.84	.42	.8
73	97.15	2.85	.34	1.58	1.27	1.2
75	98.60	1.40	.21	.85	.55	.8
77	97.41	2.59	.34	.77	1.82	.9

Application of Analyses

Number	F. F. A.	Absol. Oil	Absol. Loss	Kettle Loss	Kettle Yield	Efficiency
76	.7	96.99	3.01	6.72	94.28	92.20
2	.9	98.67	1.33	5.54	94.46	95.76
55	1.00	97.26	2.74	5.00	95.	97.70
1	1.00	98.	2.00	6.05	93.95	95.90
3	.8	98.74	1.26	4.91	95.09	96.22
73	1.2	97.15	2.85	6.57	93.43	96.17
75	.8	98.60	1.40	4.89	95.11	96.46
77	.9	97.41	2.59	5.86	94.14	96.64
Avg.	.91	97.85	2.15	5.69	94.42	96.38

Analyses and Application of 12 Savannah Refinings

Table No. 5 (c)

No.	F.F.A.	Kettle Loss	Kettle Yield	Absolute Oil	Absolute Loss	Alkali Absorp.	NaOH for F.F.A.	F.F.A.	Efficiency
39a	1.7	8.26	91.74	96.80	3.20	.49	.23	1.64	94.80
35a	1.55	6.78	93.22	97.08	2.92	.40	.22	1.55	97.02
43	.95	6.54	93.46	96.90	3.10	.34	.16	1.13	96.46
44	1.35	7.28	92.72	96.96	3.04	.42	.19	1.34	95.62
42e	1.35	7.83	92.17	98.02	1.98	.31	.18	1.27	94.06*
45b	1.00	7.68	92.32	96.68	3.32	.41	.12	.84	95.50
34b	2.65	7.56	92.44	96.11	3.89	.55	.36	2.54	95.70
40b	1.10	7.70	92.30	96.69	3.31	.47	.16	1.09	95.46
37e	3.60	8.18	91.82	94.00	6.00	.81	.51	3.60	97.68**
41a	.95	7.34	92.66	96.89	3.11	.52	.18	1.23	95.64
36a	1.25	6.42	93.58	96.82	3.18	.49	.16	1.13	96.68
38c	1.15	7.02	92.98	97.25	2.75	.46	.15	1.06	95.62
Avg.	1.55	7.38	92.62	96.68	3.32	.45	.22	1.54	95.85

*Peanut Oil.
**Cold Pressed—Also Off.

Table No. 5 (d)

	Bayonne	New Orleans	Memphis
No. Refinings	11	9	8
Average F. F. A.	1.08	1.53	.91
" Loss by Oil	5.99	6.31	5.69
" Absolute Loss	2.48	2.95	2.15
" Loss not Fat	.96	.99	1.03
" F. A. as Soap	1.53	1.86	1.11
" Kettle Yield	94.01	93.68	94.31
" Absolute Oil	97.52	97.15	97.85
" Efficiency	96.51	96.48	96.38
NaOH Absorbes	.39	.35	.28

Effect of Settling Crude Oil

Table No. 6

	1	73	76
ABSOLUTE OIL:			
Whole Sample	98.	97.15	96.99
Settled Sample	99.5	98.60	98.82
Difference	1.5	1.45	1.83
ALKALI ABSORPTION:			
Whole Sample	.25	.34	.34
Settled Sample	.18	.25	.16
Difference	.07	.09	.18
F. A. AS SOAP:			
Whole Sample	.79	1.58	1.35
Settled Sample	.60	1.32	.71
Difference	.19	.26	.64
LOSS NOT FAT:			
Whole Sample	1.21	1.27	1.66
Settled Sample	.15	.08	.47
Difference	1.06	1.19	1.19
FREE FATTY ACID:			
Whole Sample	1.00	1.20	.70
Settled Sample	.64	1.13	.64
Difference	.36	.07	.06
NaOH (Acidity No.)			
Whole Sample	.14	.168	.098
Settled Sample	.09	.16	.090
Difference	.05	.008	.008

Analysis of Cold Pressed Oil from New Orleans

Table No. 7

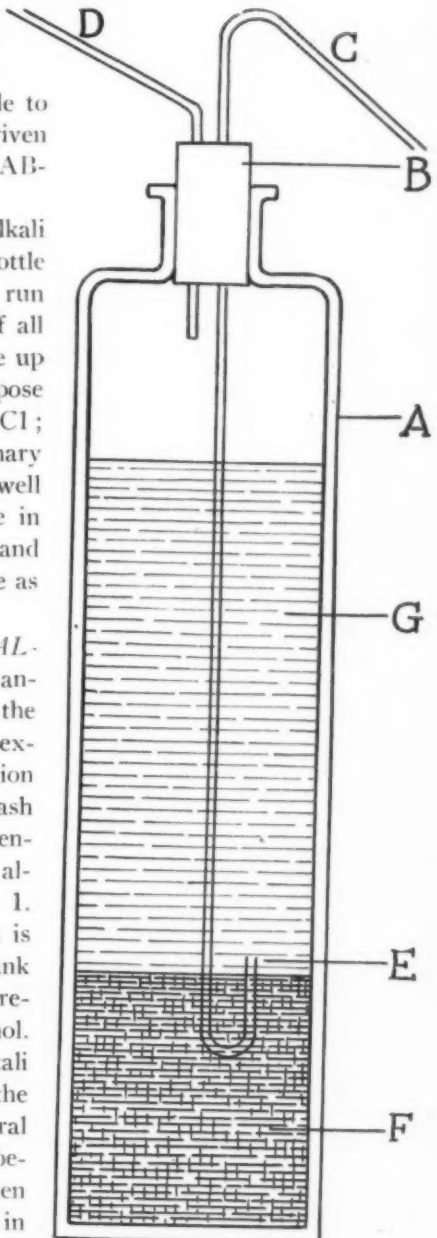
Absolute Oil	97.32
Absolute Loss	2.68
Alkali Absorption	.67
F. A. as Soap	1.05
Loss Not Fat	1.63
F. F. A.	1.48
P_2O_5	.049

Alkali absorption may not be O.K. Alcohol solution was nearly black and end point was very uncertain.

gasoline; warm over lamp till gasoline vapors are all driven off; cool and weigh. The result will be the amount of actual refined oil possible to obtain by alkali refining from a given sample of crude. This is called "ABSOLUTE OIL."

F. A. AS SOAP: Wash the alkali solution remaining in 4-ounce bottle into a beaker or evaporating dish; run down on the steam bath to drive off all alcohol and gasoline remaining; take up in 150 to 200 cc. of water; decompose with excess of dilute H_2SO_4 or HCl; collect the fatty acids as in ordinary analysis of soap-stock; wash them well on filter; extract with cold gasoline in a tared flask; drive off the gasoline, and weigh the fatty acids, reporting same as "FATTY ACIDS AS SOAP."

DETERMINATION OF ALKALI ABSORBED: Measure out another portion of the oil; treat in the same manner as for Absolute Oil, except the two washings of soap solution with 20 cc. gasoline are omitted; wash from the 4-ounce bottle into an Erlenmeyer flask, using 30 cc. of neutral alcohol, which must be at least 1 to 1. At the same time the caustic potash is added to the oil run 10 cc. for a blank into an Erlenmeyer flask and add thereto 25 cc. of the same 1 to 1 alcohol. When the titration of the excess alkali is made in the soap solution add at the same time 30 cc. of the same neutral alcohol to the blank. The difference between the quarter normal HCl taken up by the blank and that required in the soap solution is the measure of the amount of alkali absorbed by the oil. This determination should be made quickly, as the results are apt to be too



Sketch of Wash Bottle Arrangement for Making the Analysis Under Discussion.

A 4 oz. sample bottle; B cork with two perforations; C blow-off tubes with broken end at E; D blow tube; F alcohol potash soap layer; G gasoline oil layer.

high if the solutions are allowed to stand around any length of time. By titrating in an Erlenmeyer flask over a piece of white paper or a white dinner plate it is not difficult to get the end point with phenolphthalein.

Accuracy of Method

The method has been tried out thoroughly during the last two months.*

Table No. 1 shows check results on Absolute Oil on crudes taken from a variety of sources. Plenty more of similar checks can be furnished.

Table No. 2 shows the results of applying the method to refined oils. Samples marked "B" were from different refinings at Bayonne. It will be noted in looking at this table that the sum of the F. F. A. and the moisture is a remarkably close check on the Absolute Loss as shown by the method. Results given on New Orleans No. 110 and 88 oil were made before it occurred to us to find out the reason for the Absolute Loss. The 88 oil in question came from Bayonne at a time when the plant was not running properly and unquestionably contained moisture and some F. F. A., probably about .03 to .04 per cent. The New Orleans sample was taken from the refining kettle and doubtless would, if tested for moisture and F. F. A., show up about the same as the Bayonne samples.

Reporting of Results

Table No. 3 gives analyses of composite samples sent from different refineries. It also shows the way the results should be reported. The Loss Not Fat as shown on this report is the difference between the F. A. as Soap and the Absolute Loss. In order to determine the organic matter in the Loss Not Fat, the crude should be analyzed for moisture and the percentage of moisture subtracted. The NaOH absorbed shows the amount of caustic necessary theoretically to refine the oil. Below this is shown the NaOH necessary to combine with the F. F. A. The difference between these two factors shows the actual excess of NaOH necessary to combine with the organic matter and form the F. A. found as soap, which latter comes from the breaking down of complex glycerider. The F. F. A. is determined very carefully in the usual manner. The F. A. —F. F. A. shows the amount of fatty acids saponified over and above the free fatty acid, and it is necessary to saponify this much free fatty acid before the oil can be refined. This has been determined by careful experiment. The last line shows whether the color drops slowly or quickly after shaking up with alcohol in the Absolute Oil Test. We have found that when the color drops slowly an oily soap-stock or high Free Oil Loss almost invariably happens.

Further tests showing accuracy of results, are shown in Table No. 4,

* i.e., from March 22, 1917.

which gives checks on (12) Bayonne refinings, (8) New Orleans, and (8) Memphis refinings.

The application of analyses to actual refinings are shown in Table No. 5, which gives analyses and their application to refinings from different plants.

It is interesting to note, that with the prime crude worked, the average kettle efficiency was very close to 96.5 per cent. The tables show also that absolute oil and kettle efficiency are not entirely governed by free fatty acid.

Effect of settling of crude oil is shown very fully in Table No. 6, where the absolute oil is greatly increased by the settling of the crude oil samples, taken from different refinings.

Thorough settling on the three samples tested, showed 1.45 to 1.83 per cent difference between the whole sample and the well settled one. This is mostly due to the loss, not fat in the oil.

The analysis of cold pressed oil, given in Table No. 7, is interesting as showing an apparent F. F. A. of 1.48 per cent, whereas only 1.05 fatty acid would accur as soap. In other words, the gossypol titrating as F. F. A. gives an erroneous result, and free fatty acids determined in the usual way are frequently not free fatty acids at all.

Summary

It has been shown that it is easily possible to obtain the exact amount of refined oil in a given sample of crude by an accurate analytical method.

The refining efficiency in a well operated refinery is 96.5 per cent on prime crude oils.

That proper settling of crude oil results in an increase of the absolute oil of 1.5 per cent and upwards.

That the free fatty acid test may show acidity due to other causes than free fatty acids.

EXAMINATION OF TUT-ANKH-AMEN'S COSMETIC

A. Chaston Chapman, F.R.S., and Dr. H. K. Plenderleith have pointed out that the tomb of King Tut-Ankh-Amen at Luxor was opened by Dr. Howard Carter in 1922 after having remained sealed for 3,300 years, and tests made with sterile swabs, indicated the absence of bacteria within prior to opening. Among the many objects discovered was a sealed calcite cosmetic jar of peculiar design, which contained a yellowish brown fatty substance having an odor at first suggestive of cocoanut. A unique opportunity was here afforded of studying the stability of a fatty substance which had been preserved in sterility over such a long period. Chemical examination of the material showed it to be almost entirely organic in nature, consisting of (1) a fatty portion representing nearly 90 per cent of the whole; (2) a resinous portion; and (3) a small inorganic residue of calcium salts—the latter obviously derived from the action of fatty acids upon the jar. The fatty matters consisted partly of saturated acids of the ordinary fatty acid series, chiefly palmitic and stearic acids, and partly of the so-called "oxidised acids." Assuming that the latter had been formed by the oxidation of acids of the oelic acid type, the quantity found would point to the presence in the original fat of 25-30 per cent of oelin or other unsaturated glycerides. Glycerol, free and combined, still existed in the fatty portion to the extent of 5.46 per cent, which was equivalent to 4.8 per cent of the cosmetic itself. The quantity of unsaponifiable matter was very small, and it was found impossible to detect the presence of cholesterol, phytosterol or other similar crystalline substance. The bulk of the evidence seemed to suggest that the cosmetic consisted originally of about 90 per cent of a neutral animal fat, with about 10 per cent of some resin or balsam. (*Chemical Age*, August, 1926.)

QUANTITATIVE DETERMINATION OF THE "BREAK" (AND "FOOTS") IN LINSEED OIL*

By GEORGE S. JAMIESON and WALTER F. BAUGHMAN

It is believed that one indication of the quality of linseed oil is the quantity of the break—the so-called mucilaginous materials dissolved by the oil during the crushing process—and the quantity of the sediment, which is termed foots. As is well known, linseed oil gradually deposits foots during storage. These foots are a portion of the break which has precipitated. Some of the higher melting glycerides may also separate from the oil with the foots. Much thought and time have been spent by investigators in attempting to develop a satisfactory procedure for the quantitative determination of the break and foots in linseed oil.[1]

The method designated as D-51-18T[2] is still being studied by Sub-Committee V. on Linseed oil A. S. T. M. This method consists in shaking 25 cc. of linseed oil with 25 cc. of acetone and 10 cc. of an acidified saturated calcium chloride solution. The mixture is allowed to stand in a burette for 24 hours, and the volume of the precipitated break and foots (if present) is then read. The method to be described has an advantage in that it is a gravimetric procedure which can be completed in about 6 hours. Gravimetric results cannot be compared with those obtained by the D-51-18T volumetric method.

The procedure employed in the present investigation is based upon the Wesson method[3] for the determination of the total quantity of neutral glycerides in crude oils. After some practice, if close attention is paid to following the directions, this method is capable of yielding reasonably accurate results. The method is as follows:

The Method

Weigh accurately 10 grams of the sample in a 50 cc. flask and transfer with the aid of petroleum ether to a 500 cc. pear-shaped separatory funnel, using in all 50 cc. of low boiling petroleum ether (B. P. less than 80° C.). Have the stopcock of the separatory funnel lubricated with water. Agitate the oil and petroleum ether until a homogeneous solution is formed. Add 10 cc. of a 14 per cent potassium hydroxide solution, insert the stopper, and shake vigorously for 3 minutes. Then add 25 cc. of 50 per cent alcohol and shake for 15 or 20 seconds. Allow to stand until the mixture separates into 2 layers. If the alcohol-alkali solution is allowed to remain

* Presented before the Paint and Varnish Section, A. C. S., at the Philadelphia meeting, September, 1926.

[1] Glenn H. Pickard, J. Oil and Fat Ind., **2**, 60 (1925).

[2] Proc. Amer. Soc. Test. Mat. **20**, 678 (1920).

[3] G. S. Jamieson & W. F. Baughman, The Cotton Oil Press, **6**, (No. 4) 33 (1922).

in contact with the petroleum ether solution too long there is danger of saponifying some of the neutral oil. A contact of one-half hour will not cause a perceptible error and this time is more than ample to effect a good separation of the layers. Draw off the lower layer and the precipitate into a 200 cc. separatory funnel, and rinse the inside of the outlet tube of the 500 cc. separatory funnel with a little petroleum ether. Add 20 cc. of petroleum ether to the contents of the 200 cc. separatory funnel, shake, and allow the layers to separate. Draw off the lower layer and the precipitate into a 250 cc. beaker. Rinse the outlet tube of the separatory funnel with petroleum ether into the beaker. Add the upper layer to the main petrolum ether solution in the large separatory funnel. Pour the alcohol-alkali solution back into the 200 cc. separatory funnel and extract with another 20 cc. portion of petroleum ether. Repeat this treatment a third time to insure the complete recovery of neutral oil. Save the alcohol-alkali solution for the determination of the fatty acids. Wash the petroleum ether solution of the oil three times with 15 cc. portions of 50 per cent alcohol and add the washings to the alcohol-alkali solution in the 250 cc. beaker. Transfer the solution of the oil to a weighed 300 cc. Erlenmeyer flask and rinse the separatory funnel with several small portions of petroleum ether. Distill off as much as possible of the solvent by placing the flask in a water bath. Heat the flask in an oven at 120° or 125°, using an atmosphere of carbon dioxide to prevent oxidation of the oil, until a constant weight is obtained. In most cases 2 hours' heating is sufficient. Calculate the percentage of neutral oil.

Place the beaker containing the alcohol-alkali solution on the steam bath and evaporate the alcohol. Then add about 75 cc. of water and, when the soap is dissolved, acidify the solution with hydrochloric acid. Cover the beaker and heat on the steam bath until the fatty acids have collected on top of the solution. Cool until the fatty acids become solid, filter, and wash with water until the fatty acids are free from chlorides. Place the funnel containing the filter paper with the fatty acids in the 250 cc. beaker and heat on the steam bath until beaker and filter are dry. Dissolve the fatty acids with small portions of petroleum ether. If the solution of the fatty acids is turbid, refilter it through the original filter. Collect the filtrate and washings in a weighed 200 cc. Erlenmeyer flask. Remove the solvent as described for the determination of neutral oil, and weigh. Calculate the percentage of fatty acids. To obtain the per cent of break or break and foots, subtract the percentages of neutral oil and fatty acids from 100.

The results obtained are given in Table I on the page following:

Table 1

ANALYSES OF RAW LINSEED OILS

Sample	Neutral Oil %	Fatty Acids %	Break %	Acid Value of Oil	Acidity as Oleic Acid %	Iodine Number (Hanus)
A. S. T. M. No. 60* ..	98.32	1.26	0.42	2.23	1.12	179.5
" " " ..	98.35	1.22	0.43			
A. S. T. M. No. 61* ..	98.49	1.23	0.28	2.23	1.12	184.8
" " " ..	98.45	1.30	0.25			
A. S. T. M. No. 62* ..	97.92	1.43	0.65	2.28	1.14	190.5
" " " ..	97.93	1.45	0.62			
" " " ..	97.95	1.38	0.67			
No. 1 (1924)	98.36	1.15	0.49	1.89	0.95	178.5
"	98.38	1.19	0.43			
"	98.39	1.20	0.41			
No. 2	98.77	0.98	0.25	1.67	0.84	188.9
"	98.69	0.98	0.33			
No. 3	98.54	1.19	0.27	2.17	1.09	181.3
No. 4	98.97	0.77	0.26	1.26	.63	186.3
"	98.96	0.75	0.29			
No. 5	98.18	1.38	0.44	2.35	1.18	184.0
"	98.16	1.37	0.47			

* Figures given under "Break" include also the foots. Each sample was thoroughly mixed before analysis. These oils were kindly furnished by R. D. Bonney, Chairman of A. S. T. M. Sub-Committee V on Linseed Oil.

Oils 1, 2 and 3 were hot pressed and oils 4 and 5 were cold pressed.
Oils 1 and 2 were expressed from Argentine seed while 3, 4 and 5 were obtained from American seed.

Occasionally, after the withdrawal of the alcohol-alkali solution, a small quantity of a finely-divided precipitate gradually separates from the petroleum ether solution of the neutral oil. During the washing with 50 per cent alcohol, this precipitate settles between the layers and it is usually safer not to withdraw it with the wash solution as there is danger of losing some neutral oil. In the few cases observed it has been found possible to decant the petroleum ether from this slight precipitate. However, if necessary, the precipitate should be removed by filtration. Then the greatest care must be taken to recover all of the neutral oil from the filter with petroleum ether.

From the results given, it will be observed that there is apparently no relation between the quantity of break in an oil and its iodine value. The one sample with the highest iodine number also contains more of the break than any of the other samples given in the table.

It is hoped that this method will be given a thorough test by those interested in the quantitative determination of break or foots in linseed oil.

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MODERN COD LIVER OIL AS A SOURCE OF FAT-SOLUBLE VITAMINS

By ARTHUR D. HOLMES, Ph.D.

The following paper was prepared by Dr. Holmes for the *Boston Medical and Surgical Journal*. Since its appearance last April, requests for reprints have been so unusually heavy as to indicate a very widespread interest in the subject and commendation of the paper. This makes it appear desirable for the *Journal* to reprint the paper, for which it has secured the permission of the author. —*The Editor.*

Cod liver oil has been used for therapeutic purposes for centuries and until very recently it has been an extremely crude product. In general, it was separated from the liver tissues by allowing the livers to decompose until the tissues had weakened to such an extent that the oil was released from the liver cells. Then on account of its lower specific gravity, it rose to the surface and was removed by skimming. Such oil is now called "rotted oil" and appears in the trade as "Cod oil," being widely used in the leather industry. Oil prepared from livers undergoing decomposition is ordinarily of a dark color, rather viscous and of a decidedly nauseating odor and taste. The undesirable odor and taste are due to a number of secondary products such as butylamine, amalyamine, hexylamine, and dihydrolutidine, which may be produced by decaying cellular matter.

During the long period that oil of this nature was used for therapeutic purposes, many theories were developed concerning its therapeutic value. Its virtue has been ascribed to its phosphorus or iodine content, to the unusual fatty acids that were liberated during digestion, and to its value as a source of energy. At the present time, however, it is believed that cod liver oil is largely of value as a source of the essential fat-soluble vitamins. As a consequence of this conclusion, the old rule-of-the-thumb process for the production of cod liver oil was soon replaced by methods developed by science.

This change was in no small measure hastened by investigations which showed that the vitamin potency of cod liver oil was influenced materially by the nature of the manufacturing process. Obviously, many manufacturing processes were studied, and as a consequence of numerous investigations, a thoroughly modern process has been developed. This involves the use of steam kettles, specially designed separators, brine cooled presses, and other equipment designed particularly for the manufacture of medicinal cod liver oil. Thus it is now possible to produce cod liver oil of a light yellow color, of low free fatty acid content possessing a wholesome odor and flavor and possessing a high vitamin potency.

Investigations have shown that the vitamin content of cod liver oil varies over wide limits[1] and that American cod liver oil has a higher potency than Norwegian oil. Doubtless many factors contribute to the difference in vitamin potency between American and Norwegian cod liver oil, but many people believe that the higher potency of American oil is

due in no small measure to the fact that in America cod fishing continues throughout the entire year, whereas in Norway, cod fishing is largely confined to the spawning season. It has been estimated[2] that the ovaries of a 21-pound cod contains 2,700,000 eggs. It is reasonable to suppose that Nature has provided all of these eggs with every essential, including vitamins, necessary for maintenance and development until the new individual has established its dietary regime. Consequently, it is perfectly reasonable to assume that during the active stage of the reproductive cycle, the store of vitamins in the liver is materially withdrawn by the developing ova.

Early in the testing of various materials for their vitamin content, it was found that cod liver oil was the richest natural substance in vitamin A and vitamin D. Whether it contains vitamin E is still an unsettled question. The role and value of vitamins A, D and E may be summarized as follows:

Vitamin A is antiophthalmic, is required for growth and maintenance, serves to increase the body's resistance to bacterial infection[3] with pneumococcus, anthrax bacillus, and other pathogenic organisms (very recent evidence indicates that cancer[4] may result from diets low in vitamin A), and reduces the possibility of bacterial invasion of the mucous membranes of the ear and nasal cavities.[5] Vitamin D is antirachitic and is essential for all young, both human and animal (the continued use of diets low in vitamin D cause faulty bone formation as evidence by rickets, by caries, by poor tooth structure, and by that portion of defective hearing[6] due to poor ear bone development). Vitamin E, sometimes called the fertility vitamin, is essential for normal reproduction (its economic importance to poultrymen and stock raisers is evident).

For at least two or three hundred years, cod liver oil has been recognized as of particular value in the treatment of rickets. The results of numerous clinical studies combined with the results of many investigations of experimental rickets supply conclusive evidence that vitamin D is antirachitic and necessary for normal calcium and phosphorus deposition. Thus we have an explanation of why cod liver oil may be considered as a specific for rickets.

Recently it has been shown that rickets may also be satisfactorily treated by the use of sunlight or ultra-violet light. This observation raised the question as to how two such unlike substances as cod liver oil and ultra-violet light could apparently accomplish the same result.

One theory developed to account for this phenomenon assumed that cod liver oil must contain radiant energy similar to that of ultra-violet light. The report of Kugelmass and McQuarrie[7] that they had detected emanation of ultra-violet light from cod liver oil seemed to substantiate

this theory. Unfortunately, for this theory, a number of investigators[8] working at widely different points were unable to detect ultra-violet radiation from cod liver oil and recently[9] the original report has been retracted. Accordingly, the similarity of cod liver oil and ultra-violet light as regards antirachitic activity does not seem to be accounted for by the theory of radiant energy.

At the present time there seems to be considerable evidence that their similar activity is to be explained on the basis of chemical action. It has been shown that when cholesterol is irradiated with ultra-violet light, it acquires antirachitic activity. Since human fat is rich in cholesterol and is found in the skin, one naturally inquires whether the same reaction that has taken place in vitro between cholesterol and ultra-violet light producing antirachitic activity can take place in the skin when it is exposed to ultra-violet light. Theoretically, this is possible, for Macht, Bell and Elvers[10] recently have shown that the long ultra-violet rays will pass through living white human skin providing it is not exceedingly thick.

To test this theory, Hess[11] fed rachitic rats irradiated and non-irradiated calf skin and found that the non-irradiated was not effective and the irradiated cured the rachitic condition. The next step obviously was to repeat the experiment with irradiated and non-irradiated human skin and like results were obtained. It, therefore, would seem that the cholesterol of human fat residing near the surface of the body undergoes the same chemical transformation when acted upon by ultra-violet light as that taking place in vitro when pure cholesterol is irradiated by ultra-violet light.

Such evidence obviously aroused keen speculation as to the chemical nature of the antirachitic vitamin. A number of investigators found that oils containing cholesterol or phytosterol but exhibiting no detectable antirachitic value will develop antirachitic potency when subjected to irradiation with ultra-violet light. It may be noted that cod liver oil which is the richest natural source of vitamin A and vitamin D is also one of the richest sources of cholesterol. Also it has been shown that if cod liver oil is saponified, its antirachitic value will be found not in the soap nor glycerine, but in the unsaponifiable portion which is exceedingly rich in cholesterol. In view of these findings, the question arises, does cholesterol possess antirachitic value?

Hess[12] has shown that pure cholesterol when fed to rachitic rats does not improve their condition. Steenbock[13] has precipitated cholesterol from cod liver oil by digitonin and subsequently found that the oil retained its antirachitic potency. Zucker and co-workers[14] also conclude that the antirachitic substance in cod liver oil is not cholesterol, although like it in solubilities. These results indicate that cholesterol is not the antirachitic

substance in cod liver oil, but that vitamin D is evidently a definite chemical substance of sterol nature present in the unsaponifiable portion of cod liver oil and very likely associated with cholesterol.

Quite definite information is now at hand regarding the chemical composition of vitamin A. This has been separated from cod liver oil, spinach, and other sources by Takahashi,[15] who has made an exhaustive study of its composition and reactions. By saponifying a highly potent cod liver oil and subsequently separating the constituents of the unsaponifiable portion a substance was obtained which represented 0.1% of the original oil. This he named Biosterin. It was found impossible to purify this by crystallization, but purification was accomplished by distilling under pressure. At 147°—150° a 30% yield of the original substance was obtained in an apparently pure form. The composition of this was found to be $C_{27}H_{44}O_2$. In a study of the nature of the oxygen grouping, it was found that this was neither an aldehyde nor ketone grouping, but rather the oxygen atoms occurred as hydroxyl groups, one of which reacted as a tertiary alcohol. Biosterin reacted with acetic and benzoic aldehydes and with phosphorus pentachloride. It produced definite color reactions. When fed under definite experimental conditions as regards experimental animals, ration, and laboratory procedure, it was found to have a potency such that 0.001 to 0.005 mgms. daily sufficed to meet the vitamin A requirements of young albino rats. A larger amount 0.015 mgms. daily was required to effect recovery in laboratory animals suffering from not too severe vitamin A malnutrition. When injected hypodermically, a 0.125 gm. dose was found to be fatal in two hours. The fatal dose is thus something like 10,000 times the nutritional requirements.

In the practical use of vitamins A and D either as a constituent of the daily diet or for therapeutic treatment of nutritional diseases, it should be remembered that cod liver oil is without an equal as a source of these two vitamins. Sunshine and ultra-violet light when properly applied (an overdose has detrimental results) possess definite antirachitic value, but neither sunshine nor ultra-violet light have been shown to have any vitamin A value. Also those oils that have acquired antirachitic activity from being exposed to ultra-violet light are not comparable with good cod liver oil as a source of vitamin A if indeed they are comparable with it as regards vitamin D.

In general, our present information indicates that the long therapeutic use of cod liver oil is justified and explained on the basis of its high content of vitamins A and D, that with the assistance of science it is possible to produce a palatable cod liver oil possessing high vitamin A and D content; that these vitamins are definite chemical substances, and that at least one of these may be produced by the action of ultra-violet light on

cholesterol, phytosterol, and possibly other sterol-like substances. With this information available as a basis for future work, it is indeed probable that there will be a rapid accumulation of data in this most interesting field of investigation.

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NEW PLANT FOR FAT EXTRACTION BY SOLVENTS

Although the extraction of oil from seeds by solvents is theoretically very simple and desirable, solvent extraction has not obtained the vogue to which its inherent advantages entitle it. This is due to the coincidence of several disadvantages each by itself not of the utmost importance but taken together producing a total effect which has created a certain lack of favor. In fat extraction, as in every other commercial process, the net result desired is a profit. As oil extraction is fundamentally an intermittent process, various factors enter into the final cost such as the time spent in charging and discharging, the amount of labor spent in completing a cycle, the size of the individual charges, the amount of superintendence necessary, the cost of fuel, power, etc., the quality of oil obtained and the nature of the exhausted cake. In other words, the problem is essentially one of chemical engineering. Wherever it is possible to conduct the necessary operations mechanically instead of by hand labor, where the operations can be fitted in so that an efficient use of whatever labor is required is achieved, and where the method of operation is such that there are no considerable idle periods for any parts of the plant, considerable economy is obtained.

In the past, attempts toward economy have generally been in the direction of increase in unit size. A 3 or 4-ton extractor has required 6 to 12 hours for a complete cycle with a considerable period between cycles for discharging the extracted meal and recharging with fresh crushed meal. The use of labor is generally unevenly distributed so that economy is difficult, dilute solutions are also obtained which tend to complicate the process by the use of these final weak solutions to extract new charges and large volumes of solvent must be distilled to obtain the oil. In addition both the meal and the oil must be rendered free of solvent involving in some cases open steaming of the meal for several hours and dry and wet distillation in the case of the oil. This latter may in turn require the use of considerable condensing surfaces.

The battery system of operation does away with the distillation of large volumes of dilute fat solutions but the battery system can be used only for installations of considerable size. In general the use of agitators has been given up as ineffective and introducing unnecessary complications except for the purpose of assisting in the discharge of the extracted meal. All the above considerations are indicative of the economic disadvantages of attempting to run a solvent extraction plant. In addition there is the very serious disadvantage that the long period of time in which the materials are subjected to heat and moisture causes considerable hydrolysis of the fat with a considerable consequent lowering in value of the extracted oil due to the presence of free fatty acids. Prof. J. W. Hinchley in an

address before the joint meeting of the Institution of Chemical Engineers and the Oil and Color Chemists' Association at the Congress of Chemists held at London, July 23, 1926, described a new type of plant which has overcome many of these difficulties leading to an operation which is more economical of labor, fuel and power and yielding a product of such high quality that it is extremely probable that a revolution in the solvent extraction of fats will be accomplished thereby. Before describing in detail the exact structural features of the plant, a few remarks about the nature of the process will be made.

The plant is very small in size so that it never contains more than 400 pounds of material at a time although the output in 3 to 4 tons per 8-hour shift. The total time of extraction is approximately 30 minutes and the total time of steaming off the meal to free it from solvent is only 4 to 6 minutes. These rapid operations are accomplished by the peculiar method of treatment, by pre-heating the meal to nearly the temperature of the steam and by the fact that the steam has to pass through only a few inches of material instead of through several feet as in the usual type of plant. The stills are required to take care only of solvent well saturated with oil and the distillation of oil solution is continuous. The time of heating both the meal and the oil is reduced to a minimum.

It has long been understood by chemical engineers that heavy solvents such as trichloroethylene possess a great advantage in that the extraction can be made by upward flow of the solvent. As a rule by the use of a heavy solvent allowing it to rise through the meal and overflowing above to the extent of about half the volume of the meal itself and then draining a satisfactory extraction can be made. However, solvents such as trichloroethylene introduce problems of corrosion and affect the health of the workers so that they have not become popular. The plant devised by Prof. Hinchley is so small in size and of such a design that the use of special resistant metals and of a design which prevents loss of vapors and thus obviates any injury to the workers' health becomes possible.

With light solvents several flushes are required to produce satisfactory extraction. Since the amount of oil removed by each flush depends upon the ratio between the total solvent used and the amount retained by the meal after drainage considerable quantities of solvents are required in the plant. Thus for a unit treating 3 tons per charge, 7 tons of solvent will be necessary in the plant. In the equipment to be described, although the weight per charge is small, for an equal output, namely, 3 tons in 8 hours, not more than one ton of solvent would be required. These results are possible because small quantities of material are dealt with, the rate of solution of the fat is high and the removal of solvent from the meal is thorough. The solvent feed pipes are large, continuous agitation with the solvent takes place, the depth of the charge is only a few inches, and the

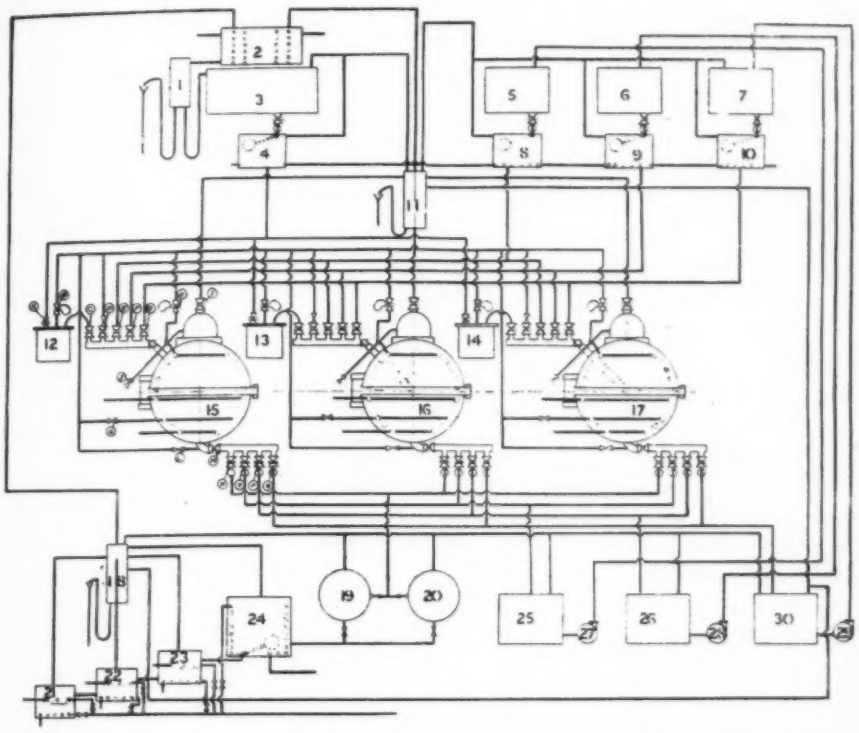

Fig. 1. Showing the Arrangement of the Plant

Drawing from Chemistry & Industry

operation is conducted in a rotating cage so that the amount of liquor left in the meal on drainage is very small. The cage or basket which contains the meal is a perforated drum the axis of which is a hollow shaft through which the steam and solvent are supplied. It rotates at different speeds in different parts of the operation. The charging is carried out by the removal and replacement of one of the end plates of the cage and the cage is inserted into a cylinder which contains the necessary gear for rotating it.

After the cage is placed in the cylinder, a valve control permits the entry of steam and the driving out of most of the air from the cylinder. Each unit consists of 3 cylinders with their cages, each one being operated separately. After the operation of charging and discharging automatically hydraulically controlled valves operated by means of a timed cam shaft control the operation of the various valves during the process. This cam shaft controls all the operations from the supply of the solvent until extraction and steaming are completed. The cam shaft makes one

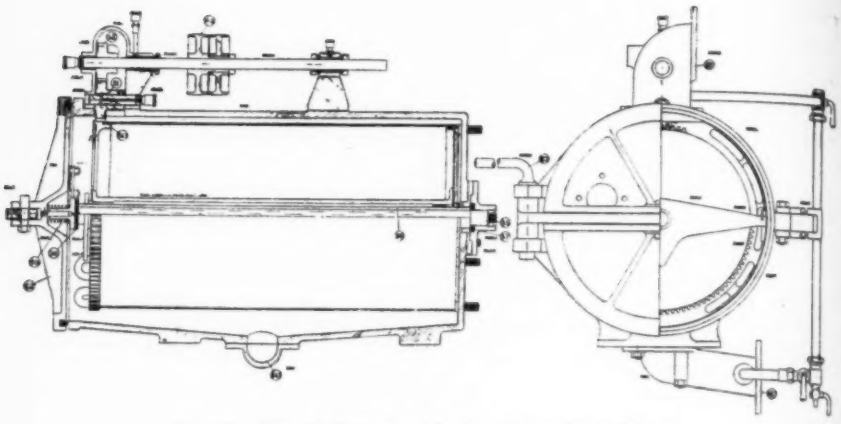

Fig. 2. The Cylinder with the Cage in Position

complete revolution in 32 minutes and has 16 movements, making each movement 2 minutes long. By means of a special control the operator, after ejecting most of the air from the cylinder as described above, throws a lever by which the cam shaft takes over the operation of the plant. At the end of the 32-minute cycle the machine stops and a signal tells the operator that the extraction is complete and that the cage containing the extracted meal is ready for removal. Although each of the 3 cylinders operates independently of the others, the solution tanks and the solutions are dealt with together. In addition to the 3 treating cylinders the apparatus contains a condenser and separator, solvent tank, solution tanks, measuring tank, solution measuring tank, and distilling equipment.

On the operation of the cam shaft the cage commences to rotate and a highly saturated solution of fat, which is called solution No. 3 enters the cylinder of the slowly rotating cage and the solution obtained runs off for distillation. No. 2 solution now enters the slowly rotating cage and is run off into solution tank No. 3. Solution No. 1 now enters and is run off into solution tank No. 2. At this point the speed of the cage is raised automatically and clean solvent enters and runs through the charge into solution tank No. 1 thus completing the extraction cycle. The cage continues to revolve at the higher speed but steam is admitted into the closed heating coils in the cylinder by which the temperature of the meal is raised to as near that of the steam as is possible. Direct steam is now admitted through the hollow shaft for 4 to 6 minutes at the end of which time the extraction cycle is completed and the apparatus stops. This method of operation produces a very highly concentrated fat solution for distillation. The highly concentrated fat solution is discharged into a regulating still which while it continuously evaporates the solvent by means of a closed steam coil converts the intermittent flow which it re-

ceives into a regular stream of liquid which enters a set of 6 small stills arranged in two parallel sets of 3 stills each. Each still contains a tray with an independent heating coil and partitions so that the liquid travels along the coil at a depth of only about 3/8 of an inch. The tray in the last pair of stills contains perforated steam coils for wet steaming and final

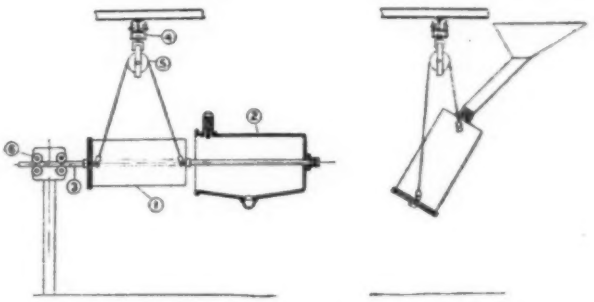

Fig. 3. How the Cages Are Charged and Discharged

removal of the last traces of solvent. The level of the liquid in the main body of each still is controlled by a constant level device by which the discharged liquid is taken from the bottom of the still while the level is maintained. The intermittent discharge of concentrated solution from the extraction plant is thus controlled to give continuous distillation and removal of the solvent from the oil. The attached figure 1 shows the arrangement of the plant. In this view the first stage of extraction is being carried out in cylinder 15. A consideration of this drawing shows how essential the automatic devices for the control of the various stages of the process are for the practical success of the plant. Obviously more cages are necessary than 3 to avoid delay during the charging period.

The operation of the machine may be considered as consisting of 7 stages:

1. Preliminary treatment of the dried meal with solvent vapor.
2. Extraction with strong solution of oil to obtain the rich solution for distillation.
3. A second treatment with fat solution which is used for operation 2 of the next charge.
4. A third treatment with dilute solution which is used for operation 3 of the next charge.
5. A final flushing with pure solvent.
6. A drying period when most of the liquid is expelled from the meal by rotation of the cage while being warmed with indirect steam.
7. Steaming with direct steam to remove traces of solvent from the meal.

During periods 1 to 5 the basket is rotating at low speed but sufficient

to keep the meal constantly turning over and changing position so that no pockets or balls are formed thus accomplishing agitation without any agitator. During periods 6 and 7 the speed of the cage is increased so that the meal forms a cylinder with a wall of even thickness and texture as a result of which the steam used for driving off the last traces of solvent is able to operate effectively without any channelling occurring. As a matter of fact the steam actually displaces some of the final solvent without evaporating it owing to the uniform texture of the meal in the cylinder. The amount of fresh solvent used for each charge is only 16 to 20 gallons.

The process described involves a system for handling solutions of different densities at varying speeds of cage rotation for which hand control would have been ineffective. Figure 2 shows the cylinder with the cage in position. The rotation of the basket is effected by means of the fast and loose pulleys on top. The cover of the cylinder is closed by a clamp device similar to that used in the ordinary horizontal gas retort. Figure 3 illustrates the method of charging and discharging the cages. At the right a cage is being charged with meal. The end plate is then clamped on and it is moved to the horizontal position shown at the left and is entered into the cylinder. A number of experimental extractions have been carried out using a complete single cylinder plant of this type. A complete 3 cylinder unit is being constructed in England and another in Germany. From the data of the experimental work it is apparent that there will be no difficulty in operating successfully with any oil seed. The data show that steam and solvent consumption will be lower than in ordinary plants and the labor costs will be less through continuous employment. The extracted meal will be discharged in a dry condition which is highly advantageous. The oil is of extremely high purity. The linseed oil obtained has puzzled even experts. It is free from the normal smell of linseed oil, when heated it becomes very pale and on boiling develops no offensive odor.

Owing to the fact that only a small quantity of solvent is required, the range of solvents is increased and differential extraction by more than one solvent becomes a commercial possibility. The results with linseed oil indicate that this method can yield a solvent extracted product equal to the pressed product and that consequently all the prejudices against solvent extracted oil must disappear.

THE MANUFACTURE OF TOILET SOAPS

By ALBERT PARSONS SACHS

The manufacture of any soap is the solution of a problem in practical fat chemistry. The problem posed is this: what chemical reaction, physical and mechanical treatment, what additions of auxiliary materials are necessary to convert a given fatty glyceride (or mixture of glycerides) into a soap of the desired chemical and physical properties (including odor, color, luster, etc.), of the requisite chemical stability (i.e., keeping qualities), while incidentally permitting the recovery of glycerine.

A fatty acid has in general the formula

$CH_3.CH_2 \ . \ . \ . \ COOH$, which may be written—FOH

in which F represents the acidyl group $CH_3.CH_2....CO$:

The formula for a simple triglyceride is then

$$C_3H_5(OF)_3,$$

and our problem is to so carry out the principal reaction:

$$C_3H_5(OF)_3 + 3NaOH = 3NaOF + C_3H_5(OH)_3$$

as to obtain the required result indicated in our first paragraph. The equation, while it represents the chemistry of our problem in a superficial manner, is by no means the complete answer to our problem, which is how is soap manufactured. As in every other industry the technic of the laboratory is merely the basis of the manufacture and is not of itself sufficient to indicate the manufacturing process. The chemist with an ordinary university laboratory training could no more produce a marketable soap than the graduate of an engineering course could with his university training alone design a battleship or a watch.

The manufacture of toilet soap is in many respects chemically the simplest soap manufacturing process known, starting as it does with the purest raw materials and yielding as its product the closest approach to pure soap which is marketed commercially. However, the physical and mechanical processes involved are quite complicated and will serve to indicate how profound an effect physical conditions have on our simple chemical reaction.

It is hardly necessary to recall the facts of soap manufacture, but this is done merely to bring out the chemistry of the processes involved. Soap manufacture consists essentially in

(1). The saponification of the fat.

(2). The separation of the soap formed from the glycerine liberated, from the impurities and the by-products.

(3). The physical treatment of the soap (including coloration and perfuming) to give it the requisite physical characteristics.

The saponification of fat is not by any means as simple an operation as the equation for the chemical reaction indicates it to be. The questions

of maximum yield of soap, maximum recovery of glycerine and the varying speeds of saponification of the oil complicate matters. Cocoanut oil saponifies very readily with the evolution of much heat, while tallow is much more difficult to saponify as are also the hardened (hydrogenated) oils. The separation of the soap from its solution is brought about by the commonly used method of organic chemistry, the salting out by the use of a salt with an ion in common, in this case common salt, NaCl, to salt out soap, NaOF. Hardened whale oil requires a brine of 1.06 specific gravity to salt out the soap formed from it, while cocoanut oil or palm-kernel oil soap requires a brine of 1.125 specific gravity for complete salting out.

In all modern soap practise mixtures or blends of fats are used as the raw material for soap. This is advantageous from several points of view. In the first place the soaps produced are better. They combine advantageous properties such as quick lathering with retention of the lather in a manner not possible for the product of a single fat. Saponification is easier in general; the hardened fats saponify much more easily when added to a partially saponified charge which causes rapid and easy emulsification; on the other hand cocoanut oil and palm-kernel oil tend to swell and boil over unless added similarly to a partially saponified charge.

Each operation in soap boiling (that branch of soap manufacture which deals with the production of the pure soap from the original fat) is known as a change. Soap production by the old cold process can actually be carried out as one step; namely, the treatment of a fat (cocoanut oil) with the requisite amount of caustic solution to produce a soap mass of the correct water content and to retain all the glycerine. This is still sometimes used by very small scale manufacturers of specialties such as shampoo pastes or even shaving creams; but it plays a part of negligible importance as compared to the great soap manufacturing industry.

On a somewhat larger scale soap boiling may be conducted with as few as two changes (strong change and fit) or three changes (brine change, strong change and fit); but the manufacture of a high grade toilet soap is a process involving six changes. They are:

1. *First brine change:* In this the charge is saponified and then grained (or salted out) by salt, allowed to settle over night and the spent lye drawn off.

2. *Second brine change:* In this the charge from the first brine change is closed (i.e., redissolved in hot water), more alkali and fat may be added, the charge is again grained with salt, allowed to settle and the spent lye drawn off.

3. *Resin change:* In this the charge is again closed, grained with salt and the resin soap added.

4. *First strong change:* In this the charge is again closed and is

opened (salted out) with caustic soda, allowed to settle and the half spent lye drawn off. This is essentially a washing process to remove impurities, especially those of color-producing character.

5. *Second strong change:* This is similar to the first strong change.

6. *Fit:* This is the final operation yielding the pure soap.

The ideal strength of caustic soda is 1.300 specific gravity. The details of quantity of water added (as such or as steam) the rate of feeding in the fat (melted, if solid at ordinary temperature) and caustic soda, the maintenance of suitable temperatures, etc., play an important role in the saponification but will not be considered in this brief review. The salt is added by sprinkling in the requisite weight while the charge or "pan" is boiling smoothly. The soap rises or grains, the brine with most of the glycerine forming the lower layer. The stronger the brine the harder is the curd or separated soap; a moderately soft curd is desirable as it permits better separation of the spent lye. The charge from the first change is settled over night and the spent lye is analyzed in the morning. If too much alkali is present the charge must be heated up again and corrected by the addition of more oil or of fatty acids. If satisfactory the sludge cock is opened and the spent lye drawn off. The sharper the separation the more successful the subsequent operations. Indeed sharp separation of the soap from the impure liquors after every change is a fundamental necessity for the production of high grade soap in good yield.

In the second change the soap is boiled up with steam, water added until the charge is closed and fairly thin. In some cases only about two-thirds of the fat is saponified in the first brine change so that the additional third and the requisite caustic are now added. Graining is produced as before, followed by settling and drawing off the spent lye.

These two changes and the resin change which follows have as their function the complete saponification of the charge and the washing out of the glycerine and impurities. The removal of all glycerine is economically impossible and the finished soap will contain 0.2-0.4 per cent of absolute glycerine.

The amount of spent lye is about equal in weight to the soap produced. The spent lye from the first change contains 4-6 per cent of glycerine, the second spent lye 2-4 per cent of glycerine, and the brine from the resin change about 1 per cent or less of glycerine. It is claimed by some that the slightest trace of resin will ultimately appear in the distilled glycerine. The spent lye from the first two changes should not ordinarily be higher than 1.105 specific gravity and will usually contain 10-15 per cent of common salt. The spent lyes are allowed to cool before being sent to the glycerine department and some soap separates out. The skimming off of this soap and its return to the process tends to increase the yield

somewhat and to prevent difficulties in the glycerine department.

For the resin change the charge is closed with as little water as possible, the necessary salt for graining is added, and then the resin as such or as resin soap. If added as resin it should be the finest water white and sufficient caustic is added to form the resin soap. Resin or resin soap is never added to a closed pan but always to a grained pan. It is added after most of the glycerine has been removed (by the spent lyes of the first two brine changes) as the resin soap even in small quantity tends to retain glycerine. When the resin soap is formed separately soda ash is used and the resin soap may be salted out once or twice to cleanse it of impurities before adding to the soap pan. After the addition or formation of the resin soap the pan is allowed to settle out. This is fairly rapid requiring only about 5 hours. As the process of graining represents driving an equilibrium to completion in one direction, i.e., the salting out of the soap, the addition of a new component, sodium resinate, may affect this equilibrium and cause the partial closing of the previously grained charge. In such a case additional salt is added to restore complete graining. The spent lye goes to the glycerine department.

The charge is now subjected to the strong changes. It is closed with water and then grained with caustic soda solution of 1.300 specific gravity. The function of this operation is to remove salt which is more harmful in subsequent operations than caustic soda and to wash the soap clean of impurities. After overnight settling the half-spent lye is drawn off. It is generally dark in color and should be used only to saponify dark colored charges (for laundry soap) as its use for saponifying fats intended for light colored toilet soaps is likely to cause darkening of the final product.

For the second strong change closing should be carried out with a minimum of water and the graining with the least possible quantity of caustic soda which will accomplish the desired result. After overnight settling the half-spent lye is drawn off to storage. If the operations have been carried out so far with care and precision the pan will be low in salt and other impurities, will contain the lowest practicable amount of half spent lye mechanically (or chemically) held in the soap and will be entirely free from unsaponified fat. It is now ready for the fit.

The fit is the most important operation in the boiling of pure toilet soaps. The desideratum is the maximum yield of pure soap. The pan is first closed by the smallest possible amount of water. The less sodium salts in solution (salt and caustic soda) the smaller the amount of water necessary. This is vitally important to a degree not required in the other changes as will be seen. Then it is grained with caustic soda in the minimum possible amount. The fitted soap separates into two layers, a bottom layer containing all the impurities (the nigre) and a top of pure

soap. So far as the fit is concerned, the nigre, which is rich in soap, is a waste product. It may be reworked for toilet soap, but should be used for darker charges if possible. A fitted soap after complete settling should contain 61-63 per cent fatty acids and up to 0.2 per cent caustic soda.

After fitting comes the cleanse or removal of pure soap. Four days at least should be allowed for complete settling of the charge and separation of the nigre, a longer time if possible. The maximum time permissible is determined by the rate of cooling (the final temperature should not drop below 150° F.); the larger pans used in modern practise permit longer and more efficient settling. The top of the fitted and settled soap is covered with a thin, hard spongy layer of "fob" which is removed by a long handled spade, leaving a clean surface of neat soap. The still hot soap is drawn off by the skimmer pipe to a feed tank, crutcher or frame.

In the manufacture of household soaps, which is similar to the above, the charge may be bleached somewhat in the fitting by the addition of a pound of zinc dust per ton of soap, but the stock used for a good toilet soap should render this unnecessary.

The yield of pure soap (as compared to total soap in the fitted charge) may be 60-85 per cent depending on the previous separation of half spent lye and the time available for the final settling. The remaining 15-40 per cent of soap in the nigre is recovered by reworking in one way or another.

The crutcher is a steam jacketed kettle with a stirrer. In this the slight excess of alkali in the soap is neutralized by the addition of cocoanut oil, clean fatty acids or sodium bicarbonate. Perfume and coloring matters are added at this stage to soaps which are not to be milled. They are always added in solution to ensure homogeneous mixture. In the older practise the soap was run from the crutcher into frames where cooling took place very slowly. Modern practise cannot permit this time-consuming and space-consuming method and has replaced it by the water cooled press type of frame. This saves time, space and labor, produces less scrap and for most purposes actually a better product. The blocks of soap from the press are cut into slabs, bars, and finally stamped into cakes. Many so-called toilet soaps receive practically this kind of processing including a surface drying, but the true toilet soap is the milled product.

A household soap should be slightly translucent and the rapid cooling tends to create just this translucency. The fact long known to soap makers has received theoretical consideration and investigation at the hands of McBain and his co-workers who have shown a physical difference to exist between the structures of rapidly cooled and slowly cooled soaps. Toilet soaps should be as opaque as possible. Slow cooling produces opacity but it is in general not permissible. The old frame cooling method yielded the best type of opaque soap.

In practically every case modern soap works cool their toilet soap for milling by running the warm soap over a series of rotating water cooled cylinders from the last of which the soap is scraped as a solid ribbon which drops on to a belt conveyor leading to the dryer. In some cases bars obtained by water cooled press processes are chipped. The product in both cases is pure soap free from fat, free fatty acids, salt and all except a few thousandths of a per cent of caustic soda.

Toilet soaps must be hard so as not to use up too quickly and this is accomplished by reducing the water content in the dryer. The best drying temperature is about 100° F., but higher temperatures are sometimes used. The final dried product should contain 80-81 per cent of fatty acids. A good toilet soap must be hard, opaque yet lustrous, uniform in texture, entirely free from material injurious to the skin. Its color (if any) should be resistant to light, its perfume should be stable and unaffected by standing. The dried flakes or chips are subjected to certain mechanical operations which will be considered later, but certain considerations may be taken up now.

The titer value of the fatty acids of the stock should not be too high; 38-40° is best, it should never exceed 40° nor drop below 37°. If the titer value is below 37° it may be necessary to dry to 82-83 per cent fatty acids to get the requisite firmness. In the case of soap flakes for sale as such the titer value is preferably a little higher, namely, 42°.

Tallow is the principal constituent of milled soaps but since the titer value is 40-46°, low titer value oils must be used in conjunction with it. For colored soaps palm oil is satisfactory, but it gives at least a creamy color to the product.

For white soaps the best white stock must be used. This includes 50-70 per cent of finest white tallow, varying amounts of cocoanut oil, lard, palm kernel oil, etc. A few per cent of castor oil tends to improve the finish of the cake. Colored toilet soaps may be made from almost any stock of the proper titer value with a color not darker than buff; no hypochlorite bleached oil must be used as this affects the dyes added and may give a chlorine odor on lathering. Half spent lyes are never used for high grade soaps as they tend to return some of the colored impurities the removal of which is one of the principal objects of the very elaborate changes of the soap boiling process. And even for colored soaps half spent lyes tend to prevent uniformity of color.

Tallow soap gives a slow but lasting lather, cocoanut oil soap a quick but very temporary lather; the mixture is ideal. Small quantities of resin tend to fix the perfume.

To return to our soap flakes or chips. As they emerge from the dryer they are chilled by a current of cold air as they must be cold when

entering the mill and cooling does not easily occur in the storage bin.

The object of milling is to crush the chips into a more or less plastic, homogeneous condition while completing the incorporation of the perfume and coloring materials. The color and perfume are added as concentrated solutions in the mixer which acts as feeder to the milling machine. Some toilet soaps are superfatted at this point. Fats may not under any circumstances be added; the superfatting is accomplished by adding 1-5 per cent of lanolin or soft petroleum jelly (melted!) during the mixing preliminary to milling. They tend to give a lustrous finish and to impart a velvety softness in use. Sometimes zinc oxide is added to the soap in the crutcher to help increase the opacity of the final product but excessive amounts tend to impart a putty-like appearance.

The mill consists of steel or granite rollers revolving at different speeds. The flakes are crushed, kneaded together again and finally emerge as homogeneous ribbons. Heating up in the mill must be prevented as rise in temperature causes loss of perfume and a diminution of opacity.

The milled soap must now be converted to cake form. This is accomplished by means of a plodding machine which is essentially an Archimedes screw squeezer so arranged as to exert great pressures. The soap is extruded as a hard continuous bar which is automatically cut into correct lengths for stamping into cake form by a suitable machine.

The compression cylinder of the plodder is water cooled to remove the heat generated which would tend to make the soap less opaque. The extrusion orifice or nozzle is gas heated so as to polish the bar which emerges.

Thus we have converted the fat into commercial soap. Numerous chemical and physical problems have not been touched on as space forbids. There is the working up of the glycerine, the production of caustic solutions, the preliminary treatment of low grade or rancid fats, the use of fillers, the addition of medicaments, the use of suitable colors and perfumes, the control methods employed throughout the soap-making process, the analysis of soap and finally that most interesting branch of chemistry, the theory of soap action.

We have considered the conversion of fat into soap, the separation of the soap from its accompanying impurities and the obtaining of a high yield whilst producing and retaining the particular kind of physical characteristics deemed desirable. The whole process is thus broadly analogous to what the chemist is always attempting to achieve in the laboratory: a high yield of pure product from a raw material available at a low price.

Domestic Exports of Oleo Oil, Tallow, Oleic Acid or Red Oil, and Other Animal Greases, Oils and Fats, N. E. S., During June

Countries	Oleo oil Pounds	Tallow Pounds	Oleic acid or red oil Pounds	Other animal greases, oils and fats, n.e.s. Pounds
Belgium	74,376	.....	.....	752,801
Germany	2,929,736	.....	.....	.....
Greece	288,689	.....	.....	28,774
Irish Free State	9,762	.....	.....	.....
Italy	.....	.....	.....	38,606
Netherlands	1,705,784	.....	.....	1,508,842
Norway	432,887	.....	.....	95
Sweden	70,219	.....	4,041	37,484
Switzerland	.....	.....	.....	89
United Kingdom	1,203,292	75,091	.....	182,625
Canada	91,712	14,394	11,550	1,079,585
Costa Rica	.....	2,300	.....	21,926
Guatemala	.....	1,668	.....	37,318
Honduras	.....	1,164	.....	6,000
Nicaragua	.....	.....	.....	8,100
Salvador	.....	.....	.....	7,980
Mexico	40	299,393	13,722	426,120
Newfoundland and Labrador	178,701	.....	.....	.....
Jamaica	74	.....	.....	.....
Trinidad and Tobago	.....	530	.....	.....
Other British W. Indies	.....	362	.....	.....
Cuba	111,584	76,279	.....	877,324
Haitian Republic	.....	120	.....	.....
Dominican Republic	.....	53,514	.....	.....
Virgin Islands of U. S.	500	.....	.....	.....
Argentina	.....	.....	.....	7,920
Brazil	28,601	.....	.....	143
Colombia	.....	17,584	.....	89,600
Dutch Guiana	.....	9,263	.....	.....
Peru	.....	.....	.....	1,368
Uruguay	.....	.....	447	.....
Venezuela	.....	.....	.....	2,995
Japan, including Chosen	11,434	.....	.....	.....
Philippine Islands	.....	.....	.....	8,036
Syria	13,791	.....	.....	.....
New Zealand	.....	.....	.....	8
Total—Quantity	7,151,182	551,662	29,760	5,123,739
Total—Value	943,988	49,218	3,092	502,668

Domestic Exports of Lard Compounds and Oleomargarine During July

Countries	Lard compounds containing animal fats		Oleomargarine of animal or vegetable fats		Vegetable oil lard Compounds	
	Pounds	Dollars	Pounds	Dollars	Pounds	Dollars
Belgium	38,205	4,203	....	....	....	....
Netherlands	19,122	2,254	....	....	....	....
United Kingdom	127,155	21,244	....	....	880	131
Canada	1,838	360	30,223	2,606	11,744	1,670
British Honduras	14,053	2,741	....	....	13,688	2,451
Costa Rica	....	....	112	26	2,676	548
Guatemala	2,645	630	....	....	16,792	3,232
Honduras	....	....	....	....	6,263	1,229
Nicaragua	8,646	1,318	....	....	1,718	294
Panama	27,295	4,436	36,520	6,234	4,698	996
Mexico	26,163	4,129	1,100	240	105,535	16,147
Newfoundland & Labrador	5,025	829	....	....	4,229	817
Bermuda	1,340	220	240	48	3,034	645
Barbados	....	....	....	....	3,780	812
Jamaica	1,300	303	400	80	5,377	1,125
Trinidad & Tobago	500	75	....	....	720	157
Other British W. Indies	18,561	3,110	12,845	2,501	21,158	3,555
Cuba	212,655	32,624	....	....	55,381	9,152
Dominican Republic	....	....	....	....	1,657	344
Dutch W. Indies	9,565	1,624	100	20	....	....
Haitian Republic	24,898	4,770	1,000	200	18,910	3,839
Virgin Is. of U. S.	12,898	2,044	3,525	672	2,615	373
Chile	11,880	2,124	....	....	9,552	1,814
Colombia	....	....	....	....	1,704	338
Ecuador	7,666	1,347	....	....	39,419	6,450
Peru	4,234	475	....	....	....	....
Venezuela	....	....	....	....	3,477	679
China	....	....	....	....	4,065	998
Japan, including Chosen	....	....	....	....	8,252	1,417
Philippine Islands	12,600	2,660	....	....	9,565	2,003
Australia	....	....	....	....	240	88
British Oceania	....	....	....	....	108	33
French Oceania	....	....	....	....	648	149
British W. Africa	3,732	653	....	....	....	....
Other French Africa	100	22	....	....	....	....
Total	592,076	94,195	86,065	12,627	357,885	61,486

Shipments from the United States to non-contiguous territories.

437—Lard compounds containing animal fats

Alaska	11,387	2,232
Hawaii	183,727	29,449
Porto Rico	4,060	783

Exports of Vegetable Oils, Expressed, from the United States During June

Countries	(Cottonseed Oil) Crude Pounds	(Cottonseed Oil) Refined Pounds	Corn oil Pounds	Cocoa butter Pounds	Coconut oil Pounds	Linseed oil Pounds	Soybean oil Pounds
Azores & Madeira Is.	.....	.....	.....	.....	.....	525	....
United Kingdom	.....	230	.....	.....	.....	.....	....
Canada	359,880	13,596	.....	32,631	1,026,976	8,683	1,106
British Honduras	.....	132	.....	.....	.....	.....	....
Costa Rica	.....	375	9,513	.....	.....	489	....
Guatemala	.....	3,332	3,400	157	.....	1,683	....
Honduras	.....	2,407	.....	.....	.....	4,019	....
Nicaragua	.....	921	750	.....	1,007	1,981	....
Panama	.....	158,963	7,650	24	2,302	4,627	....
Salvador	.....	1,875	.....	.....	1,600	675	....
Mexico	.....	34,866	72	2,490	831,328	17,898	....
Newfdld. & Labrador	.....	20,010	.....	.....	9,250	.....	....
Bermuda	.....	.....	.....	.....	.....	300	....
Other Brit. W. Indies	.....	932	.....	.....	.....	750	....
Cuba	.....	7,497	20,973	.....	58,023	23,090	3,000
Dom. Republic	.....	4,743	4,105	.....	.....	5,253	....
Dutch W. Indies	.....	3,150	1,350	.....	.....	.....	....
Haitian Republic	300	1,845	2,357	.....	.....	6,095	....
Virgin Is. of U. S.	.....	.....	300	.....	.....	81	....
Bolivia	.....	.....	.....	.....	.....	390	....
Brazil	.....	.....	.....	.....	.....	447	....
Chile	.....	.....	.....	.....	.....	563	...
Colombia	.....	3,786	4,925	240	75	15,253	....
Ecuador	.....	.....	.....	.....	1,901	.....	....
Dutch Guiana	.....	5,470	.....	.....	.....	.....	....
Peru	.....	.....	.....	280	3,590	2,567	....
Venezuela	.....	4,647	.....	.....	.....	430	....
British Malaya	.....	.....	2,450	.....	.....	.....	....
China	.....	8,050	.....	3,808	.....	10,610	....
Hongkong	.....	628	.....	.....	.....	.....	....
Japan, including Chosen	.....	38,420	.....	.....	14,090	.....	....
Kwangtung, L. terr.	.....	367	.....	.....	.....	.....	....
Philippine Islands	.....	3,345	1,380	72	.....	1,050	....
Australia	.....	.....	.....	.....	.....	1,775	....
French Oceania	.....	160	85	.....	.....	1,650	....
New Zealand	.....	1,600	5,800	.....	23,082	.....	....
British S. Africa	.....	9,372	23,034	.....	.....	.....	....
Liberia	.....	.....	.....	.....	.....	75	....
Port. E. Africa	.....	4,140	9,620	.....	.....	.....	....
Total—Quantities	360,180	334,859	97,764	39,702	1,974,230	110,959	4,106
Total—Value	35,178	52,067	14,018	14,008	201,441	14,712	538

Shipments from the United States to non-contiguous territories

Oils, Expressed

	Cottonseed		Linseed	
	Pounds	Dollars	Pounds	Dollars
Alaska	1,025	174	7,153	922
Hawaii	53,500	10,529	33,957	3,722
Porto Rico	5,519	965	69,025	8,432

ADDRESSES BEFORE THE AMERICAN CHEMICAL SOCIETY, PHILADELPHIA, SEPT. 6-10

The Composition of Corn Wax. R. L. Shriner, F. P. Nabenhauer, and R. J. Anderson. The solid material that separates when crude corn oil is chilled has been examined and found to be a wax consisting of a mixture of the myricyl esters of two fatty acids. The myricyl alcohol was isolated and shown to be identical with that obtained from beeswax. The acids were separated by fractional distillation of their methyl esters and found to be a C_{22} acid melting 76-7 and a C_{24} acid melting 85-6. The latter was shown to be n- tetracosanoic acid by means of a mixed melting point, but the C_{22} acid is not identical with n- docosanoic acid. It is apparently identical with isobehenic acid, obtained as a degradation product of lignoceric acid.

Synthetic Diets for Growth and Reproduction: Observations on the Existence of Vitamin E. (By title). V. E. Nelson and R. L. Jones. Rats were given the following diet: casein 18 per cent, salt mixture 185, 3.7 per cent, yeast at various levels from 5.0 to 16 per cent, butter fat 5 per cent and the remainder to 100 per cent was composed of dextrin. Although growth was normal, reproduction was abnormal. Animals were carried through the sixth generation on diets containing yeast as the sole source of vitamin B and butter fat as the only source of fat soluble vitamins. A large number of the females gave birth to only one litter and then ceased to have more young. The mortality of the young was high. On the same types of diets with cod liver oil at a level of 5 per cent instead of butter fat many more litters have been born and the rats have been carried so far through the third generation. The results are close to normal. The manner in which the cod liver oil is fed has a decided influence on the reproduction process. If butter fat contains vitamin E, then cod liver oil is a much richer source of this vitamin.

ABSTRACTS FROM OTHER JOURNALS

Recovery of Oils and Fats from Finely Divided Bleaching Materials or the Like. Harburger Eisen u. Bronzwerke, A. G. German Patents, 426,712, 7. 7. 21. The oil or fat contained in used decolorizing carbon or Fuller's earth may be recovered by heating the mass with a diluted solution of sodium hydroxide at 100° in an autoclave fitted with a stirring device, then raising the temperature and pressure after addition of diluted salt solution, and finally allowing the mixture to cool without agitation.

The alkali and salt treatments cause the surface tension between the solid particles of the oil and fat to be so far reduced that the two separate; the carbon or Fuller's earth settling to the bottom of the vessel, and the oil or fat forming a layer on the surface of the solution. (British Chemical Absracts B. Aug. 6, 1926.)

Further Notes on the Crystalline Bromides of Linseed and Some Other Drying Oils. Harold Toms, *The Analyst*, Aug. 1926. Gives description of experimental work and the properties of the various bodies produced, such as tetra and hexabrom stearic acids, ethyl hexabromostearate, etc.

Detection of Olive Oil Obtained by Extraction with Solvents. S. Fachini. (Giorn. Chim. Ind. Appl. 1926, 8, 178-179.)—Olive oils obtained with the help of solvents and those which have been extracted from the pressed residue and then refined are readily identified, even when present in small proportion, in olive oil obtained by pressure, by the following reaction, which is similar to Morawsky's reaction for resins. Two or three c. c. of the oil are heated in a test-tube, with an equal volume of acetic anhydride, the liquid being then shaken for a short time, cooled, and filtered through a small filter previously wetted with acetic anhydride. When brought into contact in a porcelain dish with a few drops of concentrated sulphuric acid, the filtrate soon yields a cherry-red coloration. Addition of a few c. c. of water to the product of this reaction gives a more or less intense green coloration, which gradually disappears. (T. P. H. in *The Analyst.*)

Evidence of Decomposition in Oils and Fats. J. Stamm. (Bull. Soc. Pharm. Esthonia, 1925, 5, 181; J. Pharm. Chim., 1926, 118, 214-215.)—Phenylhydrazine (2 mol.) and urethane (1 mol.) react to form diphenylcarbazide. To 10 drops of oil, or a little of the melted fat mixed with vaseline are added 5 drops of the reagent obtained by mixing 0.1 grm. of diphenylcarbazide in 10 c. c. of pure vaseline oil, and the mixture is heated for 3 minutes. Fresh oils give no coloration; a faint pink color results in the presence of 1 to 1.5 per cent. of free acidity, and if flavor and taste are satisfactory the oil is to be regarded as of good quality. If a red color results, even if the oil is organoleptically sound, rancidity will soon develop. (D. G. H. in *The Analyst.*)

ABOUT THE SMALLEY FOUNDATION CUP

The members of the American Oil Chemists' Society, especially those who have participated in the co-operative meal work, a part of the Smalley Foundation, are aware that a prize cup has been awarded each year to the collaborator having the highest average efficiency in the determination of oil and ammonia. This cup has been held by each winner for one year, and was only to become the permanent possession of the collaborator receiving it three different times. This past year the Battle Laboratory, Montgomery, Alabama, was awarded the cup for the third time, and now holds it permanently.

Dr. H. B. Battle, President of the Battle Laboratory, has kindly offered to supply a new cup, which will be a worthy successor to the first one and serve the same purpose, the same rules governing its possession as have prevailed in the case of the first one.

The members of the American Oil Chemists' Society will all unite in extending a vote of thanks to Dr. Battle for this generous offer, and also congratulate him on the excellent work which his laboratory has done in maintaining such a high standard of efficiency.

AMMONIA COMMITTEE
H. C. MOORE, *Chairman*

REGARDING CO-OPERATIVE F.F.A. TESTS

Attention is again called to the co-operative work on F.F.A. to be conducted this season by the Refining Committee of the American Oil Chemists' Society, as outlined in the August number of this Journal.

All interested are urged to send in their fee of four dollars ($4.00) without delay, as the first samples will be sent out during the month of September.